Uniaxial Tensile Properties of AS4 3D Woven Composites with Four Different Resin Systems: Experimental Results and Analysis – Property Computations

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Outline

• Introduction and Objectives
• Materials, Processes, and Weave Architectures
• Material Characterizations and Mechanical Testing
• Experimental Results and Discussion
• Material Modeling and Mechanical Property Computations
• Summary
• Acknowledgements
Introduction/ Objectives

**Composite Technology for Exploration (CTE)**
- Aimed to further advance the state-of-the-art in areas related to composite bonded joints technology
- Through case studies, the applications of composite bonded joints in heavy lift launch vehicles can reduce the mass and part counts by around 50% and 80%, respectively

**3D Woven Composites** [1, 2]
- Identified to offer good potentials in circumferential joints and end-fittings:
  - Enhanced performance (*e.g.*, delamination resistance)
  - Possibility of being woven in curved sections
  - Damage tolerance and fatigue resistance
- Known to exhibit micro-cracking
- Important to understand the evolution of micro-cracking and the influence on the 3D woven parts

**Objectives**
- Studying the evolution of micro-cracking as a function of four different resin systems, finer vs. coarser fiber yarns, and thermal cycling after processing
- Exploring how these parameters influence mechanical properties/ performance
- As an added value, taking advantage of the collected test data, modeling and computing elastic properties of the weave architectures using a finite element and an analytical technique and comparing with the test data

Material Systems

- AS4 carbon fiber with two different tow sizes (6K and 12K)
- Four different resin systems

<table>
<thead>
<tr>
<th>SN#</th>
<th>Fiber Material</th>
<th>Tow Size</th>
<th>Resin System</th>
<th>Panel / Material Designation</th>
</tr>
</thead>
<tbody>
<tr>
<td>SN001</td>
<td>AS4</td>
<td>6K</td>
<td>KCR-IR6070</td>
<td>AS4 6K/KCR-IR6070</td>
</tr>
<tr>
<td>SN002</td>
<td>AS4</td>
<td>12K</td>
<td></td>
<td>AS4 12K/KCR-IR6070</td>
</tr>
<tr>
<td>SN003</td>
<td>AS4</td>
<td>6K</td>
<td>EP2400</td>
<td>AS4 6K/EP2400</td>
</tr>
<tr>
<td>SN004</td>
<td>AS4</td>
<td>12K</td>
<td>EP2400</td>
<td>AS4 12K/EP2400</td>
</tr>
<tr>
<td>SN005</td>
<td>AS4</td>
<td>6K</td>
<td>RTM6</td>
<td>AS4 6K/RTM6</td>
</tr>
<tr>
<td>SN006</td>
<td>AS4</td>
<td>12K</td>
<td>RTM6</td>
<td>AS4 12K/RTM6</td>
</tr>
<tr>
<td>SN007</td>
<td>AS4</td>
<td>6K</td>
<td>RS-50</td>
<td>AS4 6K/RS-50</td>
</tr>
<tr>
<td>SN008</td>
<td>AS4</td>
<td>12K</td>
<td>RS-50</td>
<td>AS4 12K/RS-50</td>
</tr>
</tbody>
</table>

- Some coupons subjected to thermal cycling: -55 °C to 80 °C for 400 cycles (an 18 day process, ~1 hour per cycle)
Weave Architecture/ Parameters

- Two weave configurations proposed by Bally Ribbon Mills (BRM)
  - 3D orthogonal
  - One Z per dent arrangement

<table>
<thead>
<tr>
<th>Configuration</th>
<th>Fiber</th>
<th>Per Layer</th>
<th># of</th>
<th># of</th>
<th>Unit Cell Dims</th>
<th>% Fiber Fraction</th>
<th>% Fiber Volume</th>
<th>Z Fiber per Dent</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Warp Yarns Per cm</td>
<td>Weft Yarns Per cm</td>
<td>Warps Layers</td>
<td>Wefts Layers</td>
<td>mm</td>
<td>WARP</td>
<td>WEFT</td>
</tr>
<tr>
<td>1</td>
<td>AS4-6K</td>
<td>3.93</td>
<td>3.54</td>
<td>8</td>
<td>9</td>
<td>16.8 x 7.6 x 3.2</td>
<td>46.6</td>
<td>46.6</td>
</tr>
<tr>
<td>2</td>
<td>AS4-12K</td>
<td>3.54</td>
<td>3.14</td>
<td>4</td>
<td>5</td>
<td>19.1 x 8.5 x 3.2</td>
<td>41</td>
<td>46.4</td>
</tr>
</tbody>
</table>
Material Characterizations & Mechanical Testing

- Fiber volume fraction and void content (ASTM D3171) prior to thermal cycling
- Optical microscopy and X-Ray Computed Tomography (CT) prior and after thermal cycling

- Mechanical Testing (in **Warp direction**) at National Institute for Aviation Research (NIAR):

  Tension (ASTM D3039) with strain gages and DIC, Compression (ASTM D6641) with extensometer, Short Beam Shear (ASTM D2344), and Single Shear Bearing (ASTM D5691) with extensometer and DIC

  - Room Temperature Ambient (RTA)
    - As-processed (AP)
    - Thermally cycled (TC)

  - Elevated Temperature Wet (ETW)
    - AP
Measurement Results: $V_f$

- BRM estimated $V_f$ for 6K and 12K weave architectures to be 50.9% and 52.6%, respectively.
  - Nominal thickness of 3.175 mm vs. as-built thickness (3.175 mm to 3.327 mm)
- Consistent: $V_f (12K) > V_f (6K)$

<table>
<thead>
<tr>
<th>Panel</th>
<th>Resin</th>
<th>% Void Content</th>
<th>% Fiber Volume</th>
</tr>
</thead>
<tbody>
<tr>
<td>SN001</td>
<td>KCR-IR6070</td>
<td>~ 0, 0.2</td>
<td>47.3, 0.3</td>
</tr>
<tr>
<td>SN002</td>
<td>KCR-IR6070</td>
<td>~ 0, 0.4</td>
<td>50.6, 0.8</td>
</tr>
<tr>
<td>SN003</td>
<td>EP2400</td>
<td>1.4, 0.2</td>
<td>49.7, 0.5</td>
</tr>
<tr>
<td>SN004</td>
<td>EP2400</td>
<td>1.1, 0.4</td>
<td>51.5, 0.9</td>
</tr>
<tr>
<td>SN005</td>
<td>RTM6</td>
<td>0.4, 0.3</td>
<td>47.4, 0.3</td>
</tr>
<tr>
<td>SN006</td>
<td>RTM6</td>
<td>~ 0, 0.5</td>
<td>48.4, 1.2</td>
</tr>
<tr>
<td>SN007</td>
<td>RS-50</td>
<td>1.1, 0.2</td>
<td>47.3, 0.6</td>
</tr>
<tr>
<td>SN008</td>
<td>RS-50</td>
<td>1.2, 0.1</td>
<td>48.6, 0.9</td>
</tr>
</tbody>
</table>
Micro-cracking Assessment: Optical Microscopy

- Micro-cracks developed in all panels likely during curing process and cool down
- Micro-cracking observed mainly near the Z-fibers
- Density of micro-cracks increased after thermal cycling
  - In addition to Z-fibers vicinities, cracks distributed within the material, including individual fiber tows
- Developing an imaging technique to measure the cumulative volumes of the micro-cracks within these samples is an ongoing work at NASA
Micro-cracking Assessment: X-Ray CT

Warp-Z plane cross-section for SN001 a) AP and b) TC material

Weft-Z plane cross-section for SN001 a) AP and b) TC material
Micro-cracking Assessment: X-Ray CT

Warp-Z plane cross-section for SN006 a) AP and b) TC material

Weft-Z plane cross-section for SN006 a) AP and b) TC material
Test Results: Tensile Testing (DIC Data)

- Slightly higher stiffness and strength for 6K weave configurations (finer and tighter weave structure)
- Slight change (drop) in stiffness and strength as a result of thermal cycling
Test Results: Tensile Testing (Strain Gage)

- Higher standard deviation in modulus data attributed to strain measurements using strain gages (~9 mm x ~5 mm) and large RUC (16.8 x 7.6 mm and 19.1 x 8.5 mm) of the materials
- Standard deviations in modulus values using extensometer vs. gage data for ETW tests further support above hypothesis
Test Results: Compressive, SBS, and Bearing Strength

- 6K weave configuration consistently performed better
- ~50% reduction in strength (compression vs. tension)
- Higher standard deviation in compression anticipated to raise from narrower and shorter coupon geometry in ASTM 6641 vs. ASTM 3039
Material Modeling Approaches: FE Based

- **Finite element (FE) based approach**
  - Digimat FE

![Weave Design Model](image1)
![RUC Geometry](image2)
![RUC FE Mesh](image3)

*Matrix elements not shown*
Material Modeling Approaches: MSGMC

- **Multiscale Generalized Method of Cells (MSGMC)**
  - Developed by NASA GRC
  - Semi-analytical (efficient)
  - Provides homogenized, nonlinear constitutive response of composites

MSGMC RUCs and sub-cells across an arbitrary number of length scales

MSGMC 3D orthogonal woven representation
### Property Computations

- **AS4/RTM6 material system was selected for modeling and analysis**
  - Both 6K and 12K
  - RTA

<table>
<thead>
<tr>
<th>Configuration</th>
<th>Method</th>
<th>Material Parameter/Property</th>
<th>Material Parameter/Property</th>
<th>Material Parameter/Property</th>
<th>Material Parameter/Property</th>
</tr>
</thead>
<tbody>
<tr>
<td>AS4 6K/RTM6 (SN005)</td>
<td>MSGMC</td>
<td>61.977</td>
<td>60.096</td>
<td>9.257</td>
<td>0.059</td>
</tr>
<tr>
<td></td>
<td>Digimat-FE</td>
<td>61.833</td>
<td>59.907</td>
<td>9.691</td>
<td>0.056</td>
</tr>
<tr>
<td></td>
<td>%Δ</td>
<td>-0.2</td>
<td>-0.3</td>
<td>4.7</td>
<td>-5.1</td>
</tr>
<tr>
<td>AS4 12K/RTM6 (SN006)</td>
<td>MSGMC</td>
<td>56.803</td>
<td>57.018</td>
<td>8.885</td>
<td>0.059</td>
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<tr>
<td></td>
<td>Digimat-FE</td>
<td>56.400</td>
<td>57.500</td>
<td>9.308</td>
<td>0.052</td>
</tr>
<tr>
<td></td>
<td>%Δ</td>
<td>-0.7</td>
<td>0.8</td>
<td>4.8</td>
<td>-11.9</td>
</tr>
</tbody>
</table>

- Good agreement between Digimat-FE and MSGMC
- Largest difference of 12% for v12
Computed Properties vs. Test Data

- Only test data available: Warp (E_{11})
- Models and analysis did not include (effect of) micro-cracks

<table>
<thead>
<tr>
<th>Condition</th>
<th>Panel#</th>
<th>Strain Gage (from 5 Tests)</th>
<th>DIC (from 2 Tests)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>E_{11} (GPa)</td>
<td>SD</td>
<td>% Δ to MSGMC</td>
</tr>
<tr>
<td>RTA (AP)</td>
<td>SN005</td>
<td>57.6</td>
<td>3.3</td>
</tr>
<tr>
<td></td>
<td>SN006</td>
<td>53.5</td>
<td>4.7</td>
</tr>
<tr>
<td>RTA (TC)</td>
<td>SN005</td>
<td>57.9</td>
<td>4</td>
</tr>
<tr>
<td></td>
<td>SN006</td>
<td>48.1</td>
<td>3.4</td>
</tr>
</tbody>
</table>

- Analysis over-predicted (averaged) test values (Strain measurement technique (strain gage vs. DIC) made a difference)
- Possible sources for the differences:
  - Ignoring micro-cracks
  - Not accounting for irregularities in weave pattern (idealized modeling)
Summary

• Eight 3D woven composite panels were fabricated and subjected to material characterizations and testing
  – The 3D orthogonal weave included 6K and 12K yarn configurations and four different resin systems

• Optical microscopy and X-Ray CT revealed the presence of micro-cracks in the as-processed materials
  – Thermal cycling increased micro-crack density in all eight panels

• No significant change in tensile performance of the materials as a result of thermal cycling or ETW environment (Fiber dominated, warp direction)

• Analysis over-predicted the test results by ~5% to ~13% for the AP materials and the difference increased as the material underwent thermal cycling
Acknowledgements

• X-Ray CT support by Grace Fischetti and Dr. Justin Jones from Materials Branch at NASA Goddard Space Flight Center

• 3D weave design and fabrication by Mr. Hakan Gokce and Mr. Leon Bryn at Bally Ribbon Mills, Inc.